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Eucalyptus fibres as reinforcing materials for polypropylene based composites

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Abstract

Lignocellulosic fibres deriving from eucalyptus wood have been used as reinforcement for two polypropylene matrices: a conventional isotactic polypropylene (iPP) and a maleate modified one (iPPMA).

Before mixing, the fibres were subjected to some different extraction procedures: conventional alkaline treatments and the innovative steam explosion process.

Afterwards the composites, obtained by compression moulding, were thermally, morphologically and mechanically characterised.

1. Introduction

According to their high specific properties, low cost, biodegradability and renewability, the vegetable fibres can be utilised as reinforcements for plastic materials [1, 2]. The possibility of these fibres to act as good reinforcement agent are dependent on their cellulosic content, fibre size and a strong fibre/matrix interfacial adhesion [3, 4, 5].

In the present work vegetable fibres deriving from *Eucalyptus Occidentalis* trees have been used to reinforce polypropylene matrices. The choose of this lignocellulosic material is due to the fact that the eucalyptus plants are largely available in Mediterranean lands ; moreover its utilisation today is very poor and partially limited only for the production of cellulose pulp.

The validity to use lignocellulosic fibres as reinforcements for polypropylene (iPP) has been demonstrated in some previous papers [6, 7]. Particularly, a modified polypropylene matrix, obtained by inserting a small amount of maleic anhydride, was demonstrated to well interact with vegetable fibres, especially if they have undergone a strong cellulose extraction.

In order to obtain good lignocellulosic fibres we have utilised different extraction procedures : some conventional alkaline treatments and the innovative steam explosion technique [8]. This latter is a physical process applied on a wooden structure able to separate the lignocellulosic materials into its main component : cellulose fibres, lignin and hemicelluloses. Fibres obtained through this process are known very reactive due to their large surface area [8].

Thus, the aim of our work was the achievement of new composite materials polypropylene/eucalyptus fibres that can be used in those applications where lightness, biodegradability, good properties and low cost are required.

2. Materials

Two polypropylenes were selected as matrices (kindly supplied by Montell): a polypropylene (iPP) whose trade name is H30S, and a maleate one (iPPMA) whose trade name is Hercoprime G211.

The eucalyptus wood by "Istituto per la Valorizzazione del Legno-Firenze" of the National Council of Research (CNR) were supplied.

Four different pre-treatments on the fibres were tested in order to achieve cellulosic fibres. The details of the treatments in Table 1 are described.

3. Techniques

In order to achieve the composites, the materials (polypropylenes and eucalyptus fibres) were mixed in a Rheocord apparatus at a temperature of 190°C for ten minutes; the maximum rate speed was 16 RPM. Successively a grinding mill equipped by a 2mm mesh milled the composites. Sheets for mechanical testing were prepared, according to the standard ASTM D256 and ASTM D638, by compression moulding at 60 bar at 190°C for ten minutes. The fibre content was 20% by weight for all the composites. The obtained composites are listed in Table 2.

Successively, the specimens prepared in order to study the mechanical behaviour were characterised by tensile test (head speed 10^{-3} m/sec), Charpy impact test (head speed 1m/sec; Ceast Autographic Pendulum) and dynamic mechanical test (DMTA) in bending mode (strain <1%; frequency 1Hz; Scientific Lab apparatus).

The processing parameters (Time and temperature) were selected by means the thermal characterisation on all the materials by a Thermogravimetry (TGA) device (Mettler M3, processor TC11) and a Differential Scanning Calorimeter (DSC) (Mettler DSC30).

Finally a morphological analysis was performed by Scanning Electronic Microscope (SEM) (Apparatus Hitachi H30) on the tip of the fracture surface of the samples.

4. Results

In first column of Table 3, the amount of residual extracted material obtained by using the three alkaline procedures are shown. Of course the most part of extracted material should be composed by neat cellulose and thus it seems that procedure EU1 is more able to extract an high amount of cellulosic fibres. This result is in agreement with the data reported in the last column, where the highest value found of water adsorbed content indicating an higher swelling degree of the eucalyptus fibres. The degradation temperatures of alkaline treated fibres, obtained by thermogravimetric analysis, are shown in column 2 and not very differences seem to be among the different extractions. Anyway these degradation temperature values are enough high in order to process the fibres at the polypropylenes melting temperatures (180°C).

The results of the tensile tests carried out on the composites prepared both with iPP and iPPMA matrices in Table 4 are reported. An increase of the modulus (E) up to 20% were found. Moreover the iPPMA based composites show values of the strength at break (σ_b) higher than the neat matrix, indicating a strong fibre/matrix interfacial

adhesion ; while the iPP based composite shows a strong decrease of the strength with respect of neat matrix. All samples show a brittle mode fracture and therefore a low value in elongation at break (ϵ_b). Finally the composite reinforced by USEP fibres show an higher value of σ_b and a low modulus : this could be explained by the fact that, although a strong adhesion between the EUSEP fibres and the iPPMA matrix was obtained, less improvement of mechanical properties is observed owing to significant damage of the structure of the eucalyptus fibres during the steam explosion process.

The glass transition values, for all examined samples, measured by DMTA, in Table 5 are shown : it can be pointed out that the fibres seem to have no influence on polypropylene Tg.

The values of the impact test parameters, the critical stress intensity factor (K_{Ic}) and critical strain energy release (G_c) in Table 7 are shown; all the composites present an improvement of both G_c and K_{Ic} . However this improvement is much higher for iPPMA based composites. The EUSEP show a poor performance as filler with respect of EU1 and EU2.

Figures 1a-1b show the SEM micrographs of the two matrices used. Deformation areas, "mackerel" of different size owing to the nucleating agent by maleic anhydride are clearly visible. The sample iPP/EU3 shown in Fig. 1c, presents a poor adhesion between three filler and the matrix. No whitening, indicating stress occurred near the interface is visible.. On the contrary the sample iPPMA/EUSEP (see Fig. 1d) show a better fibre matrix adhesion indicating the stress that the matrix has transmitted to the fibres.

Work is in progress to optimise the steam explosion parameters in order to obtain better performances for the eucalyptus fibres.

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